

# **Polyimide Composites Properties of RTM370 Fabricated by VARTM**

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# High Temperature Polyimide Composites Materials & Processing

- ◆ Conventional PMR-15, PMR-II-50, AFR-PE4 polyimide composites all require solvent-based prepregs for part fabrication ⇒ *time consuming, costly and the use of solvents and diamines are hazardous*
- ◆ Fabricate net-shape polymer matrix composites from resins in the melt via RTM or VARTM using preforms ⇒ *eliminate costly hand lay-up and hazard*  
⇒ *produce 30% cost saving & 20% weight saving for complex parts*  
⇒ *adaptable to automatic process*

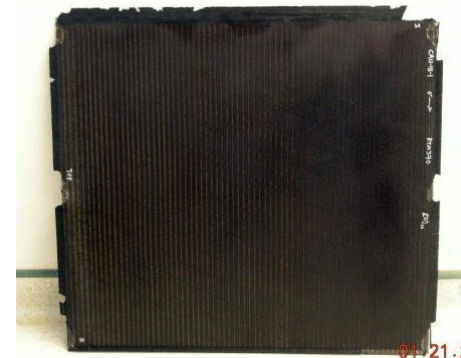
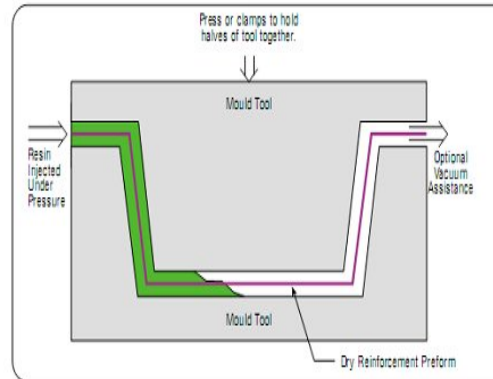
**Challenge:** RTM or VARTM requires low-melt viscosity that conventional polyimides derived from commercially available s-dianhydride ( $> 10^3$ - $10^5$  poise) cannot meet

- ◆ a-BPDA based imide resins have shown to exhibit low-melt viscosity (10-30 poise) at 280 °C ⇒ *amenable to low-cost RTM or VARTM process*
- ◆ Advance PMC temperature capability to 260-315°C beyond the state-of-the-art RTM resins, such as epoxy (177 °C) & BMI (232 °C)

# Objectives

- ◆ **Fabricated composite panels with RTM370 imide resin (~10-30 poise) by vacuum assisted resin transfer molding (VARTM)**
- ◆ **Compare mechanical properties of VARTM panels to RTM panels at 288-315°C (550-600°F)**

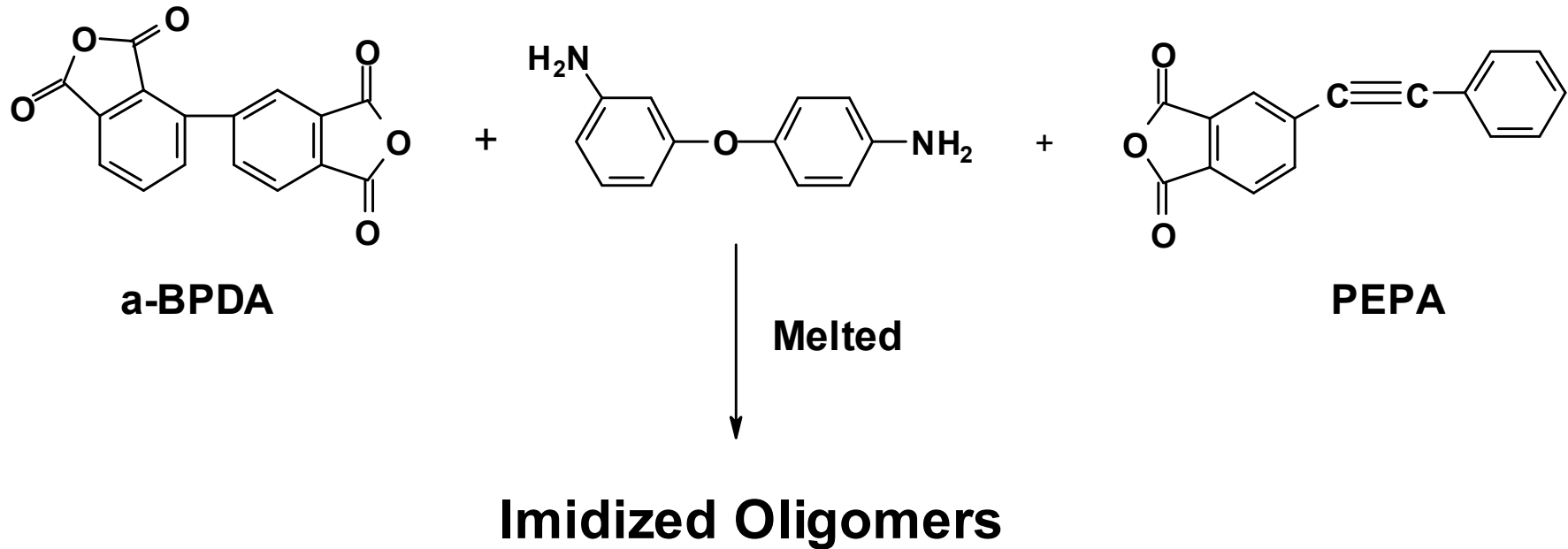
# VARTM vs RTM



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VARTM	RTM
Use vacuum bag	Need a mold (expensive)
Use vacuum only	Use pressure and/or vacuum
15-20 psi	200 psi
Suitable for large part	Suitable for small part/mold

# RTM370 Imide Resins



## ***Advantages of imide resins containing a-dianhydrides:***

- ***Lower melt viscosities***
- ***Higher  $T_g$ 's***

## ***Solvent-Free process:***

- ***No organic volatiles (Green)***
- ***Adaptable to reactive extrusion (cost saving)***

# Physical Properties of Imide Oligomers/ Resins Based on a-BPDA and 4-PEPA

Resin	Diamine	Oligomer Min. $\eta$ @280 °C by Brookfield <sup>1</sup> (Poise)	Oligomer Min. Complex [ $\eta$ ] <sup>*</sup> @280°C <sup>2</sup> (Poise)	Cured Resin T <sub>g</sub> (°C) NPC <sup>3</sup> byTMA <sup>5</sup>	Cured Resin T <sub>g</sub> (°C) PC <sup>4</sup> @ 650°F
RTM370	3,4' -ODA	8.8	6.5	342	370 <sup>5</sup>
RTM370 Composite		---	---	338 (DMA) <sup>6</sup>	350 (DMA) <sup>6</sup>

**3,4' -ODA = 3,4' -Oxydianiline**

<sup>1</sup> Absolute viscosity measured by Brookfield Viscometer at 280 °C.

<sup>2</sup> Complex viscosity measured by Aries Rheometer, using parallel plates.

<sup>3</sup> NPC = No Post cure

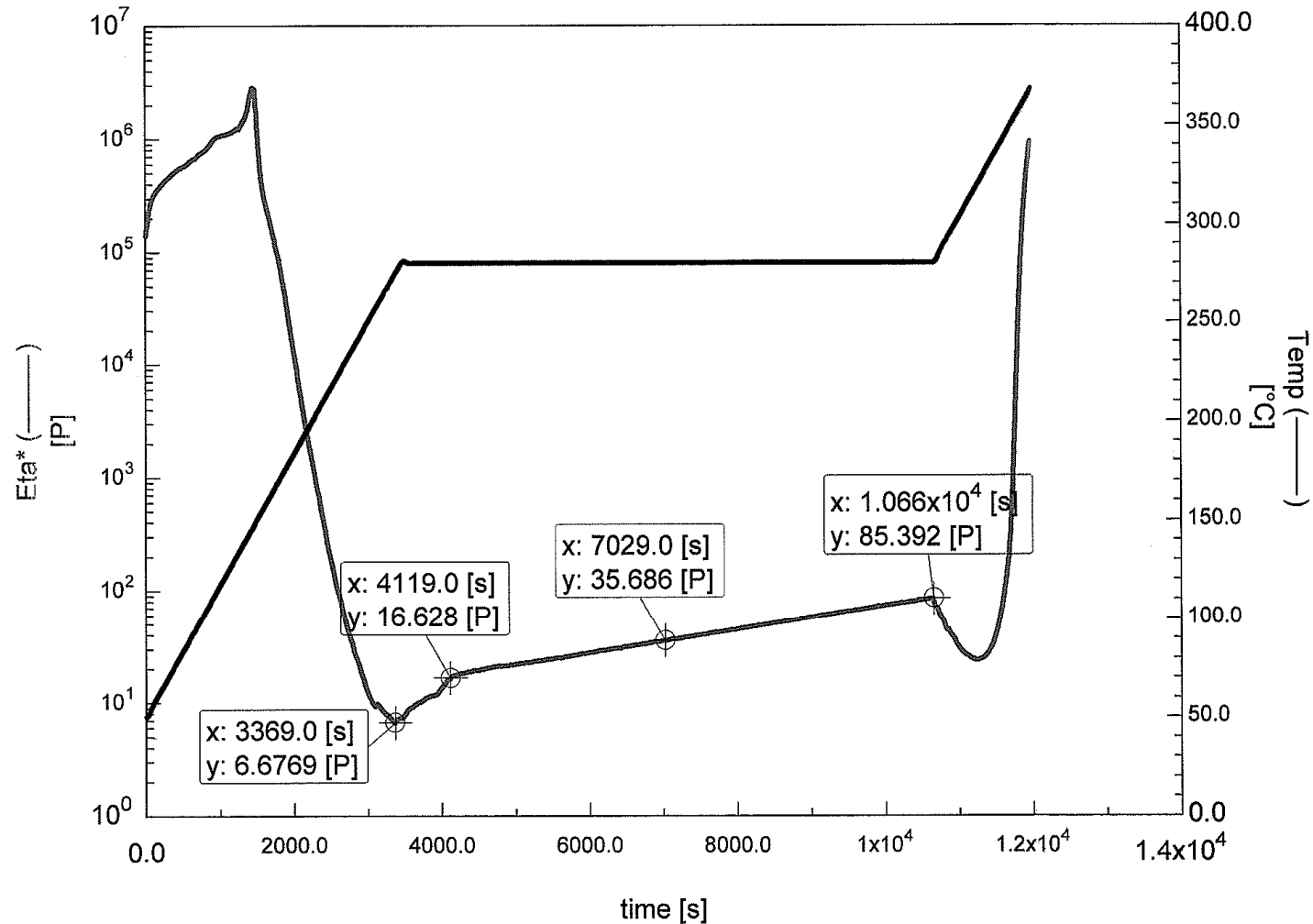
<sup>4</sup> PC = Resin Postcured at 343 °C (650 °F) for 16 hrs while composite postcured@650°

<sup>5</sup> TMA =Thermal mechanical analysis heated at 10 °C/min, using expansion mode.

<sup>6</sup> DMA = Dynamic mechanical analysis were performed at 5 °C/min heating rate,  
using single cantilever.

# Rheology of APS's RTM370 Imide Resin

## 2 hr hold at 280 °C



# RTM370 Composite Property Comparison

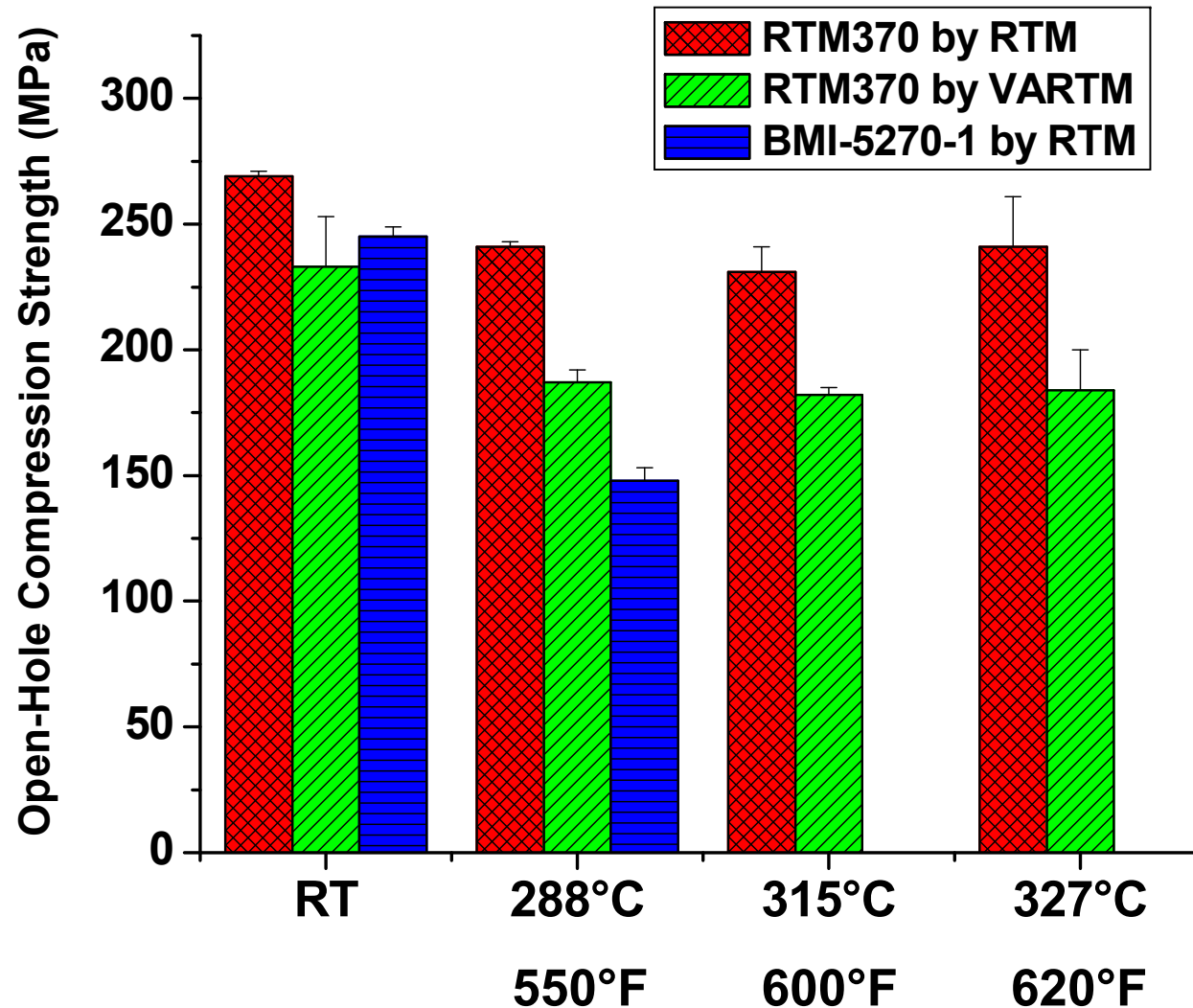
## VARTM vs RTM (T650-35/HT sizing)

	Test Temp.	OHC Strength (MPa)			OHC Modulus (GPa)			SBS Strength (MPa)		
		Initial	500h @550°F 288°C	1000h @550°F 288°C	Initial	500h @550°F 288°C	1000h @550°F 288°C	Initial	500h @550°F 288°C	1000h @550°F 288°C
<b>VARTM</b>	RT	233	194	120	37	38	33	43	37	22
	288°C	186	197	135	40	42	38	31	27	17
	315°C	182	---	118	42	---	38	29	26	15
	327°C	184	---	---	41	---	---	30	---	16
<b>RTM</b>	RT	269	287	230	44	47	44	51	54	43
	288°C	242	244	198	48	44	45	41	41	41
	315°C	231	---	---	46	---	---	31	---	---
	327°C	241	---	---	48	---	---	30	---	---



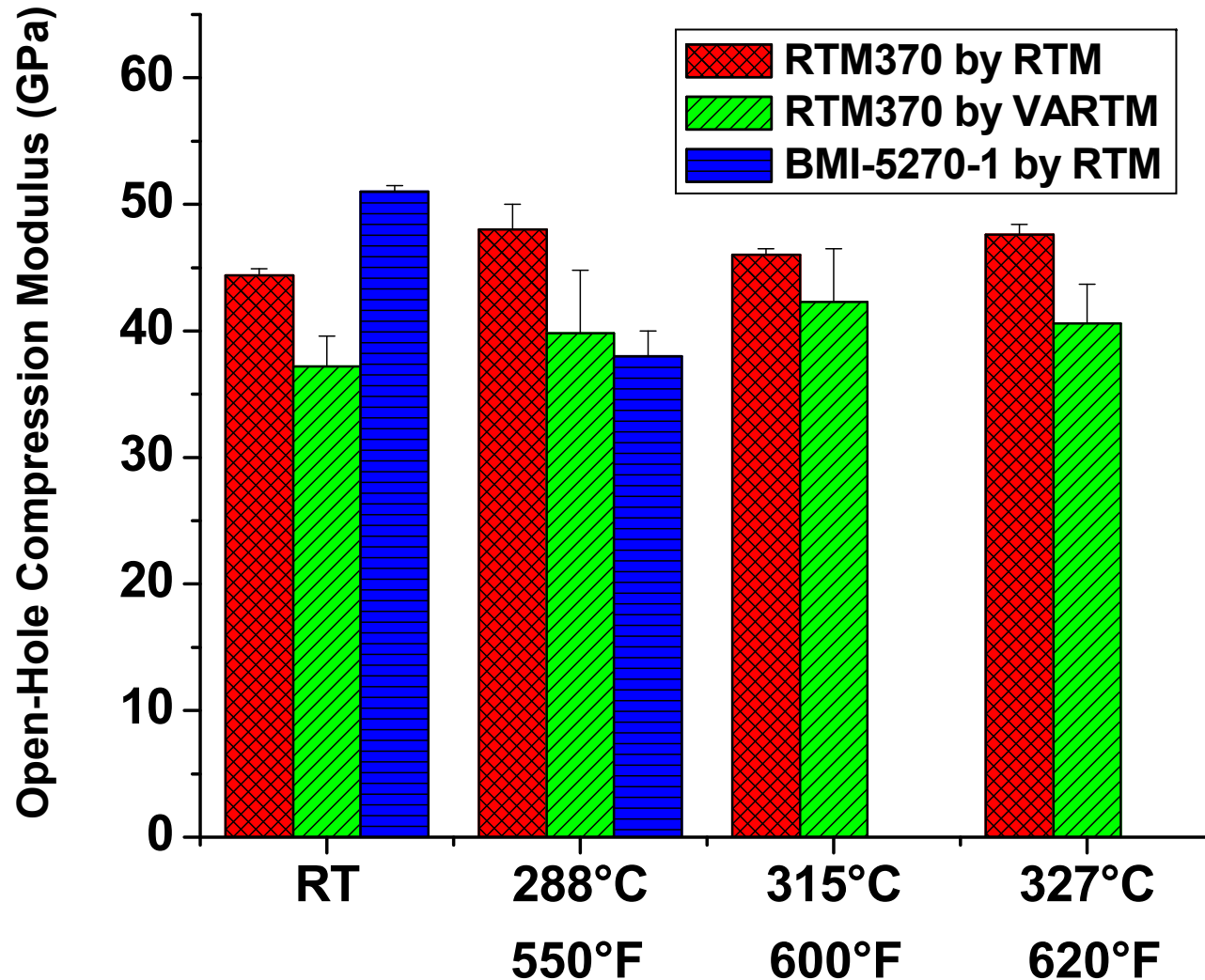
# RTM370 OHC Strength VARTM vs RTM

(T650-35/8HS/HT Sizing)



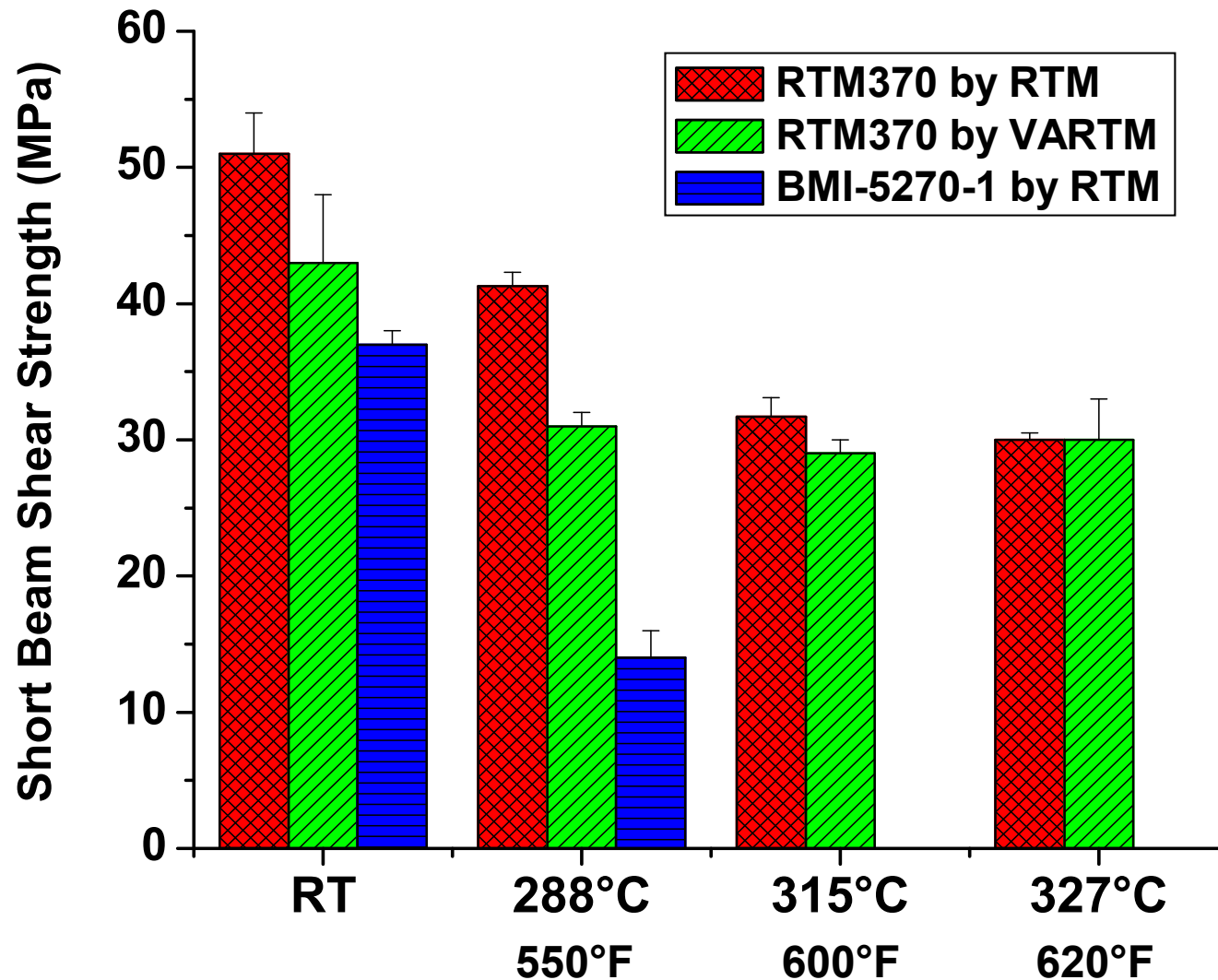
# RTM370 OHC Modulus VARTM vs RTM

(T650-35/8HS/HT Sizing)

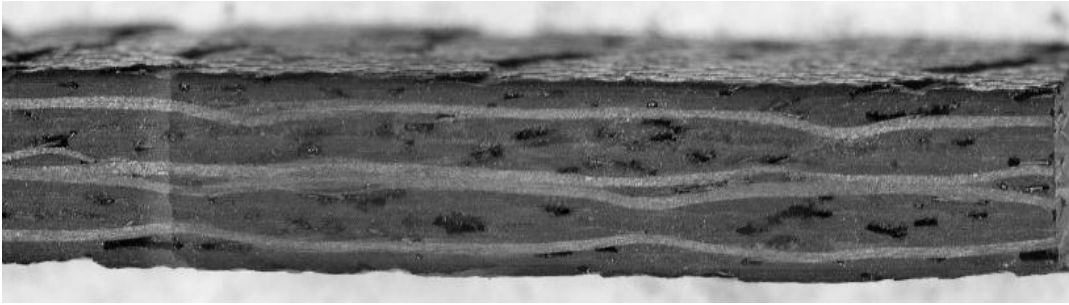


# RTM370 SBS Strength VARTM vs RTM

(T650-35/8HS/HT Sizing)



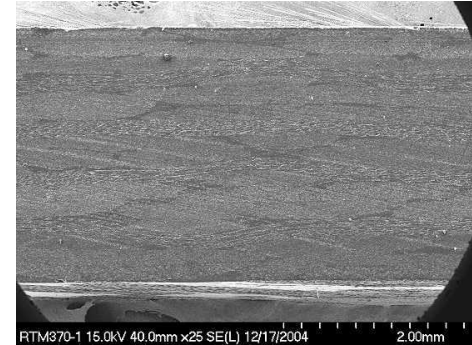
# VARTM vs RTM



SEM of RTM370 made by VARTM

Void content = ~6.5% after postcured at 650°F/8 h

Resin vs fiber content = 47-50% vs 53-50%



RTM370 Made by RTM

Void content = ~1%

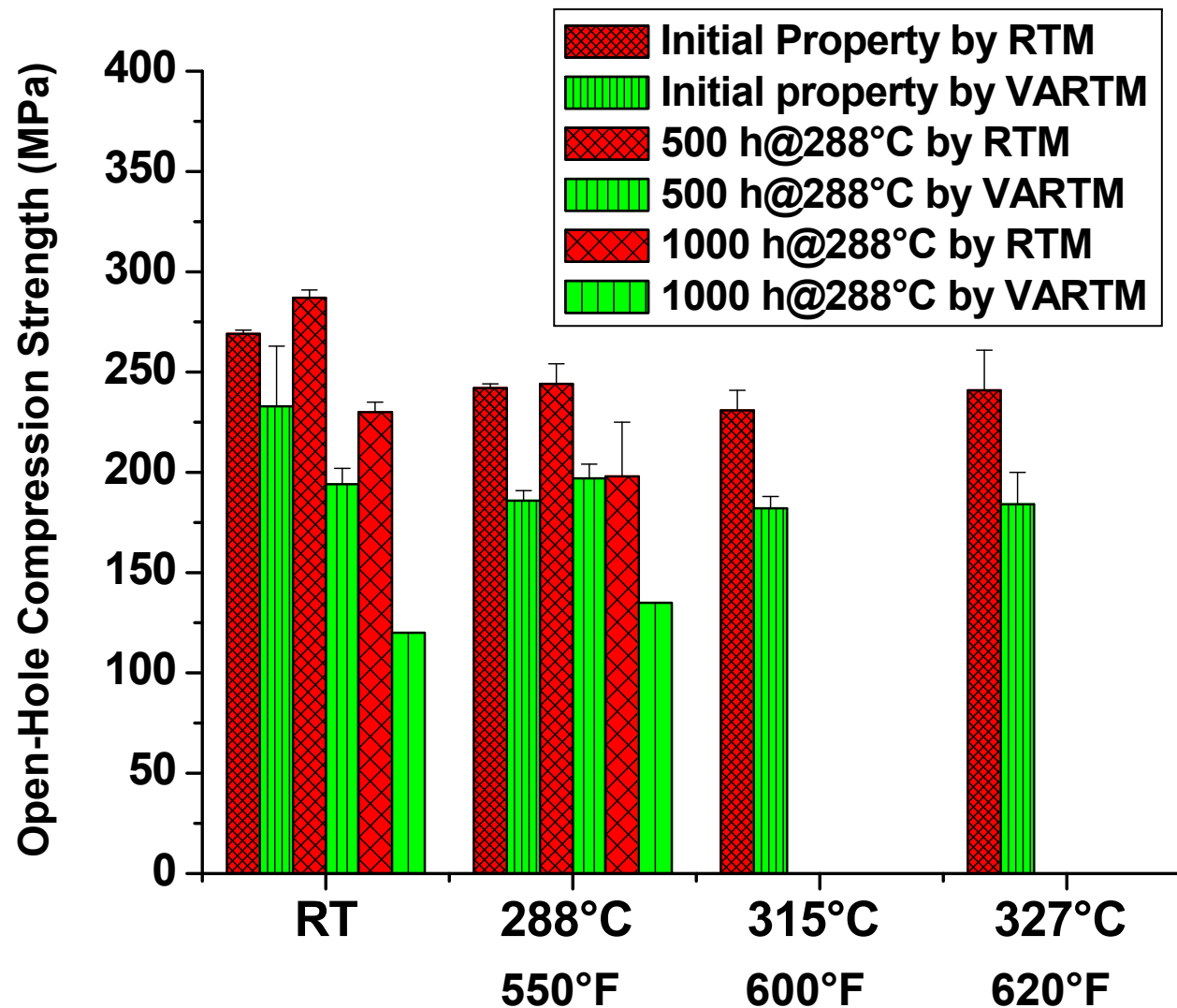
Resin content = 44-47%

VARTM	RTM
15-20 psi	200 psi
Higher void content	Lower void content

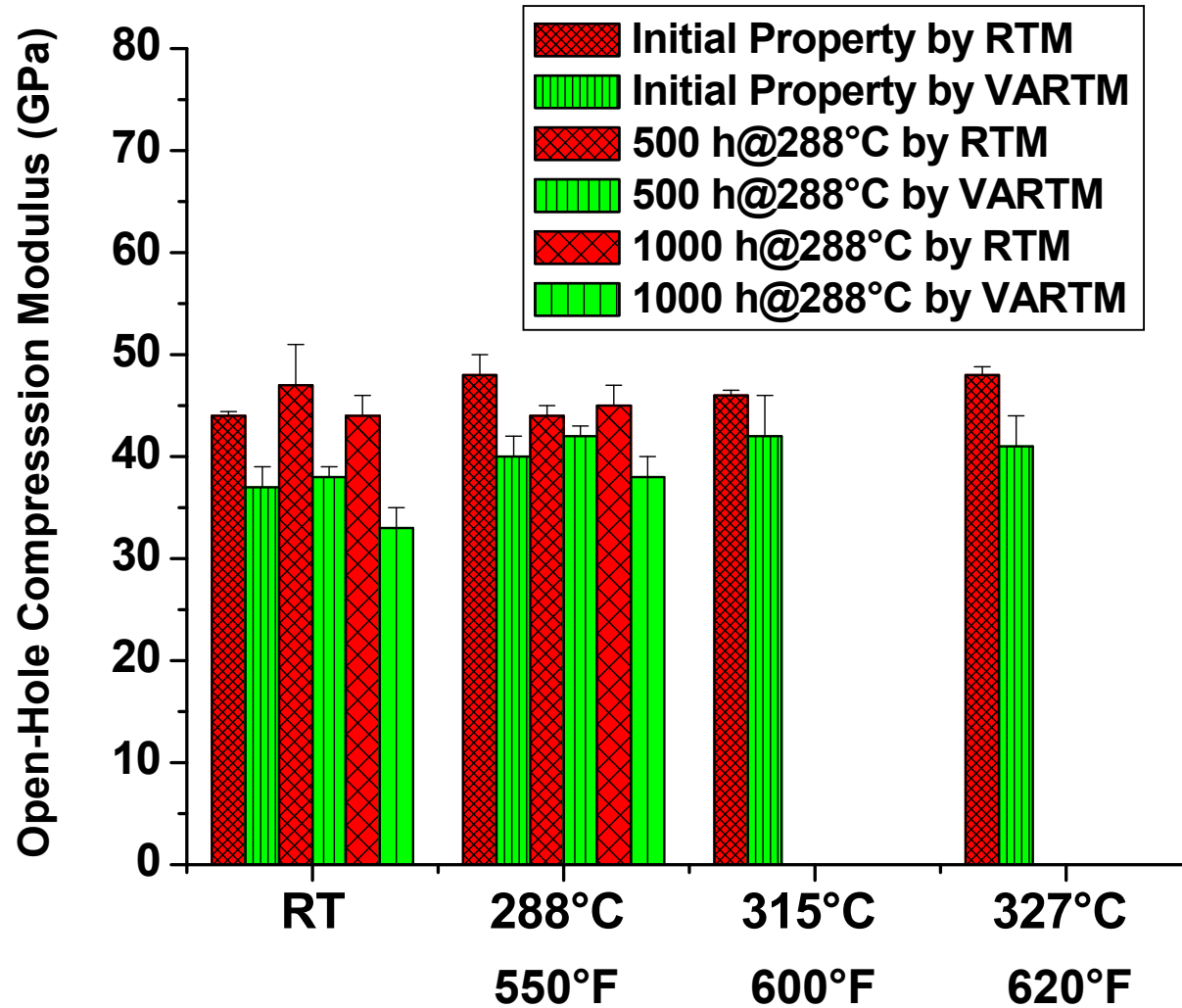
## ***Process Improvement:***

- ♦ Injection at 260°C instead of 280°C ⇒ Longer pot-life, but longer injection time
- ♦ Add hold time at ~300°C, instead of direct ramp from 280°C to 371°C

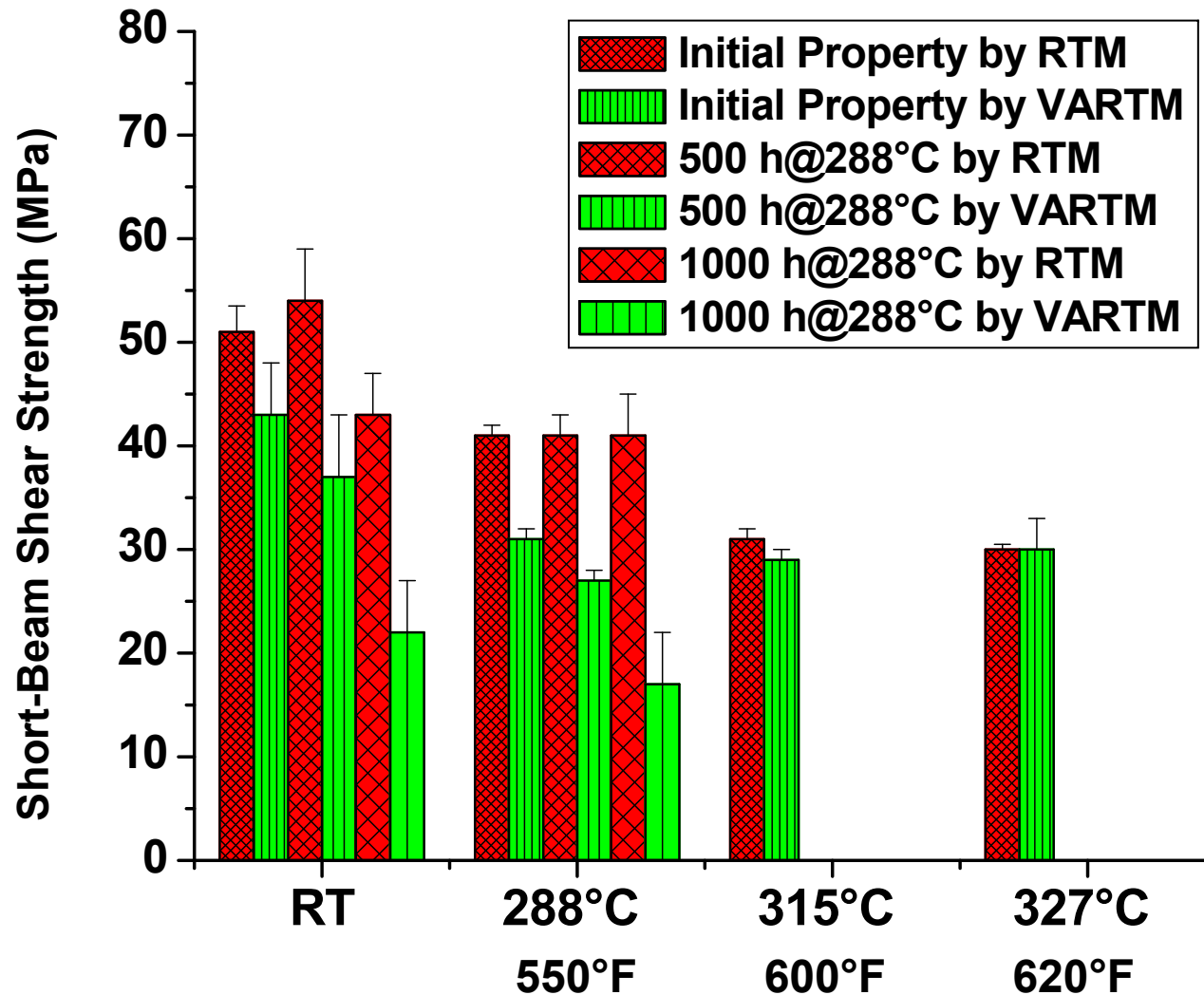
# Open-Hole Compression Strength of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



# Open-Hole Compression Modulus of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



# Short-Beam Shear Strength of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



# Summary

- ◆ Demonstrated RTM370 imide resin can be processed by VARTM out of autoclave
- ◆ VARTM panels have higher void content than those made by RTM, due to lack of pressure  
⇒ VARTM panels appeared to have slightly lower OHC, but comparable SBS at high temp. & retained good mechanical properties after aging
- ◆ Need process development to reduce void content to  $< 2\%$  for aerospace applications
- ◆ Need to improve the resin content of VARTM panels from 47-50% to 35-40%.



# **Acknowledgements**

- ◆ **NASA Glenn Tech Transfer Fund**
- ◆ **Akron Polymer Systems for supplying RTM370 resin**
- ◆ **Ohio Third Frontier Funding (Maverick)**
- ◆ **NASA Co-Op Grant to Clark Atlanta U.**
- ◆ Linda McCorkle (OAI) : SEM, Rheology  
Acid Digestion
- ◆ Brian Shonkwiler(CAU): Mechanical Testing
- ◆ Dan Scheiman (ASRC): Thermal Analysis